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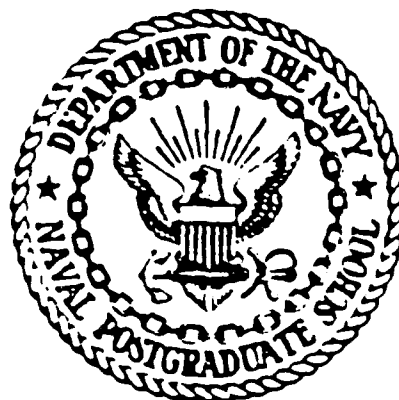
THESIS

DEVELOPMENT OF METHODS FOR
LOW TEMPERATURE DIFFUSION BONDING

by

Paul A. Muffler

September 1987

Thesis Advisor   K.D. Challenger

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An apparatus for low temperature diffusion bonding of dissimilar metals has been developed. Experiments varying the bonding temperature at constant pressure and time were conducted utilizing type 316 stainless steel samples coated with a layer of silver in order to verify the design of the apparatus and the bonding procedures. The Scanning Electron Microscope (SEM) was used to illustrate the effects of temperature variation on the microstructure of the bonds produced. The results showed diffusion bonds of silver to silver to be possible at all three temperatures used. Recrystallization and or grain growth in the silver coating occurred at the bond interface at 300°C and the entire coating was beginning to recrystallize and undergo grain growth at 400°C after 30 minutes at 20,000 psi (137.9 MPa). The experiments successfully demonstrated the capability of the apparatus, but much more development of the bonding procedure is required.

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Development of Methods for
Low Temperature Diffusion Bonding

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Lieutenant, United States Navy
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ABSTRACT

An apparatus for low temperature diffusion bonding of dissimilar metals has been developed. Experiments varying the bonding temperature at constant pressure and time were conducted utilizing type 316 stainless steel samples coated with a layer of silver in order to verify the design of the apparatus and the bonding procedures. The Scanning Electron Microscope (SEM) was used to illustrate the effects of temperature variation on the microstructure of the bonds produced. The results showed diffusion bonds of silver to silver to be possible at all three temperatures used. Recrystallization and or grain growth in the silver coating occurred at the bond interface at 300°C and the entire coating was begining to recrystallize and undergo grain growth at 400°C after 30 minutes at 20,000 psi (137.9 MPa). The experiments successfully demonstrated the capability of the apparatus, but much more development of the bonding procedure is required.

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I. INTRODUCTION

Welding (fusion or pressure) and brazing are the most common processes used to form a metallurgical bond between metals. Diffusion Bonding (also referred to by the American Welding Society (AWS) as Solid State Bonding, Diffusion Brazing, or Diffusion Welding) is an example of a pressure welding process which is gaining widespread industrial application as a viable alternative to fusion welding.

In diffusion bonding, metallurgical bonds are made by heating and pressing two similar or dissimilar surfaces together establishing interatomic bonds across the interface. The International Welding Institute (IWI) adopted a definition as proposed by N. F. Kazakov [Ref. 1]:

> "Diffusion bonding of materials in solid state is a process for making a monolithic joint through formation of bonds at atomic level as a result of closure of the mating surfaces due to local plastic deformation at elevated temperature which aids the inter-diffusion at the surface layers of the materials being joined."

The stages of conventional diffusion bonding are: (1) Materials to be diffusion bonded are pressed together. Initally the contact area is small due to surface asperities, then the pressure causes asperities to deform increasing the surface contact area. (2) Interdiffusion across the interface occurs and (3) Finally recrystallization takes place. Diffusion across and along the interface is essential to form a metallic bond between the metal specimens. In order for the diffusion to occur, the interfaces must be in contact and the atoms must have enough energy to cause mass transport across and along the interface. The requirements can be met by application of pressure which increases surface to surface contact and by increasing the temperature which raises the atomic mobility.

This thesis research involves the development of a diffusion bonding system which can perform low temperature diffusion bonding. Low temperature is defined as 0.3 to 0.5 times the melting temperature (Tm° Kelvin) of the material to be bonded. The reason for the emphasis on low temperature is so that the process being developed for diffusion bonding can be used to join materials where their properties are altered by elevated temperatures. An example of a temperature sensitive material is a material which has a metastable microstructure at room temperature. These same materials

have specific characteristics which can be lost with increases in temperature when metastable microstructures transform into stable microstructures with completely different characteristics. Another positive factor for diffusion bonding is that dimensional tolerances are maintained which eliminate costly post bonding machining or reshaping. This thesis presents a discussion of the apparatus and procedures developed, sample microscopy, a discussion of the results obtained for the effects of temperature variation, conclusions and recommendations.

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II. BACKGROUND INFORMATION

Diffusion Bonding is a simple process and has been used to join a variety of similar and dissimilar metals that cannot be fusion welded or brazed because of metallurgical incompatibility [Refs. 2-6]. For ideal or perfect samples, they need only be brought together in order to form a spontaneous joint. Despite the simplicity of the diffusion bonding process, no sample surfaces are perfectly ideal and real surfaces are not clean or perfectly flat. Real surfaces are covered with contaminants and oxide films. Carefully machined specimens are still rough with many microscopic asperities. The typical machining practice produces asperities 0.3 to 1 micron (12 micro in. to 40 micro in.) high [Ref. 1].

Surfaces must be as clean and smooth as possible on a microscopic scale in order to promote successful bonding. However, once a surface is cleaned and machined smooth all low and high spots instantaneously acquire an oxide film and contaminants. In diffusion bonding it is essential to not only remove the surface oxides and contaminants, but to also keep the surfaces free from recontamination. Two different processes are used to try and prevent the recontamination of the sample surfaces: (1) diffusion bonding in a vacuum or a controlled atmosphere, and (2) coating the sample surface substrates with an intermediate layer.

Diffusion bonding in a vacuum system requires that the samples be cleaned, heated, and pressed together inside the vacuum system. The vacuum required for diffusion bonding is usually 10^{-1} to 10^{-3} Pa (7.5×10^{-4} to 7.5×10^{-6} Torr) [Ref. 1], however, maintaining the system that supplies this vacuum can be a technical problem. Even very high vacuums 1.3×10^{-4} to 1.3×10^{-6} Pa (9.75×10^{-7} to 9.75×10^{-9} Torr) can not be considered totally inert. Small amounts of inert gases present will not readily absorb on or dissolve in metals [Ref. 7] however, impurities in the inert gases can dissolve or form surface oxide contamination. The diffusion bonding unit can be designed to be fairly simple utilizing a vacuum or an inert gas, but the commercially available units are not easily modified once designed for a specific function.

The second process uses an intermediate layer deposited on the sample surface interface. Examples of coating layers that have been used are silver, nickel, copper, and gold [Ref. 9]. The intermediate coatings used in diffusion bonding can be applied

by either electroplating or vacuum deposition. Electroplating has been used, but nearly all current efforts involve vacuum deposited intermediate layers. Once the intermediate layer has been deposited bonding may be performed at atmospheric pressure and in air. Overviews of the coating processes can be found in the literature [Refs. 8-10]. Work done by Knowles and Hazlett [Ref. 11] indicated that the diffusion bonding atmosphere (vacuum, air, oxygen, or helium) had no effect on the bond strengths of aluminum joints that were coated with an intermediate layer of silver.

All other factors considered, the success or failure of either of the two diffusion bonding processes depends on three variables. These variables are temperature, pressure, and time. The bonding pressure should be sufficient to aid in the deformation of the surface asperities. The bonding time is variable. Increases in the amount of bonding time are beneficial up to the point where grain growth impairs bond strength [Ref. 1].

For the purpose of this research, it was determined that the vacuum diffusion bonding process was impractical. The process whereby samples are coated with an intermediate layer and then diffusion bonded was the choice for this research. Samples of type 316 Stainless Steel utilizing a layer of silver were used to validate the low temperature diffusion bonding system developed. The samples to be diffusion bonded were bonded in air at a constant pressure and time utilizing an intermediate layer of silver. The bonding temperature was the only independent variable. Temperature variations were used to determine the effects of temperature on the development of low temperature diffusion bonding.

III. EXPERIMENTAL

A.   EQUIPMENT

1. Sample

The samples to be bonded are standard ASTM Tensile Specimens (Figure 3.1). Standard tensile test samples were used to facilitate easier tensile testing upon completion of bonding. Samples were machined in half to enable the surfaces to be prepared and coated. Type 316 stainless steel was chosen for the sample bonds. The chemical composition limits for type 316 stainless steel are listed in Table 1; the limits listed are shown as weight percent maximum unless shown as a range [Ref. 12].

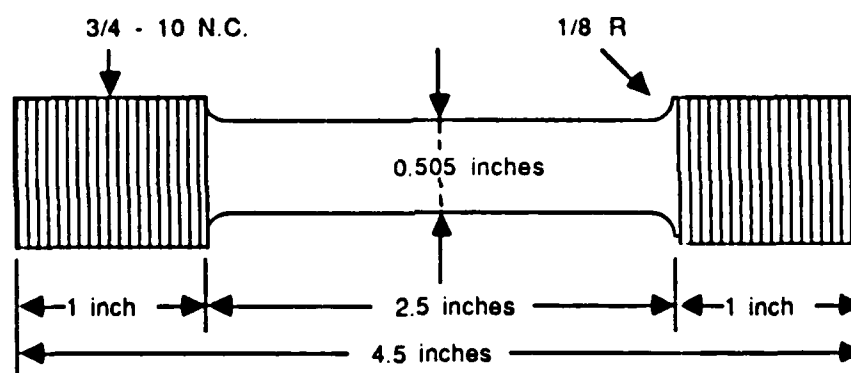Figure 3.1   ASTM Standard Tensile Test Specimen.

a. Surface Preparation

The 316 stainless steel samples were conventionally machined in half prior to the surfaces being coated with silver. No efforts were made to improve or modify the surface finish beyond that which was produced by the conventional machining process. Cleaning the machined surfaces is very important for the adhesion of the

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TABLE 1

STAINLESS STEEL CHEMICAL COMPOSTION LIMITS

Alloy 316 (content, wt%)

```
Carbon......................................0.08
Phosphorus.................................0.045
Sulfur.....................................0.030
Manganese....................................2.00
Nickel...............................10.0 - 14.0
Chromium.............................16.0 - 18.0
Molybdenum.............................2.0 - 3.0
Nitrogen...............................0.10 max
Iron.................................remainder
```

silver coating. The procedure used for cleaning the samples after the conventional machining was:

1. Wash in hot (80° Centigrade) Alconox soap and water solution

2. Rinse with tap water

3. Soak for 10 minutes in Trichloroethylene

4. Soak for 10 minutes in Acetone

5. Soak for 10 minutes in Ethanol Alcohol

6. Air dry

b. Specimen Coating

A layer of silver was sputter coated on the 316 stainless steel samples in a high vacuum diffusion pumped system at the Naval Weapons Center, China Lake, California. Sputter etching was used prior to coating in an effort to remove residual atmospheric contaminants and produce an atomically clean surface.

The work of O'Brien, Price, and Olson [Ref. 4] found that the tensile strength of the bonds formed were a function of the joint thickness. The maximum tensile strength was achieved with a joint thickness of 25 microns (1 mil). For this research, this same thickness of silver was to be used on all samples.

2. Diffusion Bonding Apparatus

The purpose of this research was to design and construct a diffusion bonding apparatus to study low temperature diffusion bonding. The diffusion bonding apparatus (Figure 3.2 and 3.3) consists of:

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1. Diffusion Bonding Press
    a) Stainless Steel Support Frame
    b) Enterpac Model RC-102 Hydraulic Ram
    c) Dura Model AISI 316 Bourdon Pressure Gage
    d) Celesco Model BCR 50B Digital Pressure Transducer
    e) TRW Engineering Hydraulic Hand Pump

2. Induction Heater
    a) Cycle-Dyne Model A-30 Induction Heater
    b) MTS Model 924.85 Temperature Control Panel
    c) Cycle-Dyne Cooling Water Pump
    d) Newport Model 267B Digital Thermocouple Readout
    e) Spiral Pancake Coil

a. Diffusion Bonding Press

The diffusion bonding press (Figure 3.4) was designed to be able to bond ASTM tensile specimens that had been machined in half and coated with a layer of silver. The pressure used on the samples was supplied by the hydraulic hand pump and the piston cylinder. The pressure was monitored by either the bourdon pressure gauge or the digital pressure gauge. The actual pressure on the system was less than the pressure at the interface of the samples. The difference in the pressures was caused by the difference in amount of surface area in the ram piston and the surface area of the samples. The area of the ram is 1.767 in^2. The surface area at the interface is 0.2 in^2. As an example, if 20 ksi (137.9 MPa) was desired at the interface, that would require the system pressure on the hydraulic ram be approximately 2265 psi (15.6 MPa). A special note is the maximum allowable pressure on the hydraulic ram or system is 8960 psi (61.8 MPa). This maximum system pressure correlates to a maximum pressure at the interface of these specimens equal to approximately 79 ksi (551 Mpa).

b. Induction Heater

The Cycle-Dyne Radio Frequency (RF) induction heater (Figure 3.5) was chosen for the development of low temperature diffusion bonding because: (1) It provides a relatively fast heat up time. (2) It produces predictable temperature gradients and (3) Modifications to the system are only a matter of interchanging the working coil [Ref. 13]. Samples used for low temperature diffusion bonding tend to form an oxide layer upon prolonged heating. The shorter heating of the induciton heating method reduces the chances of an oxide layer forming on the surface. For this
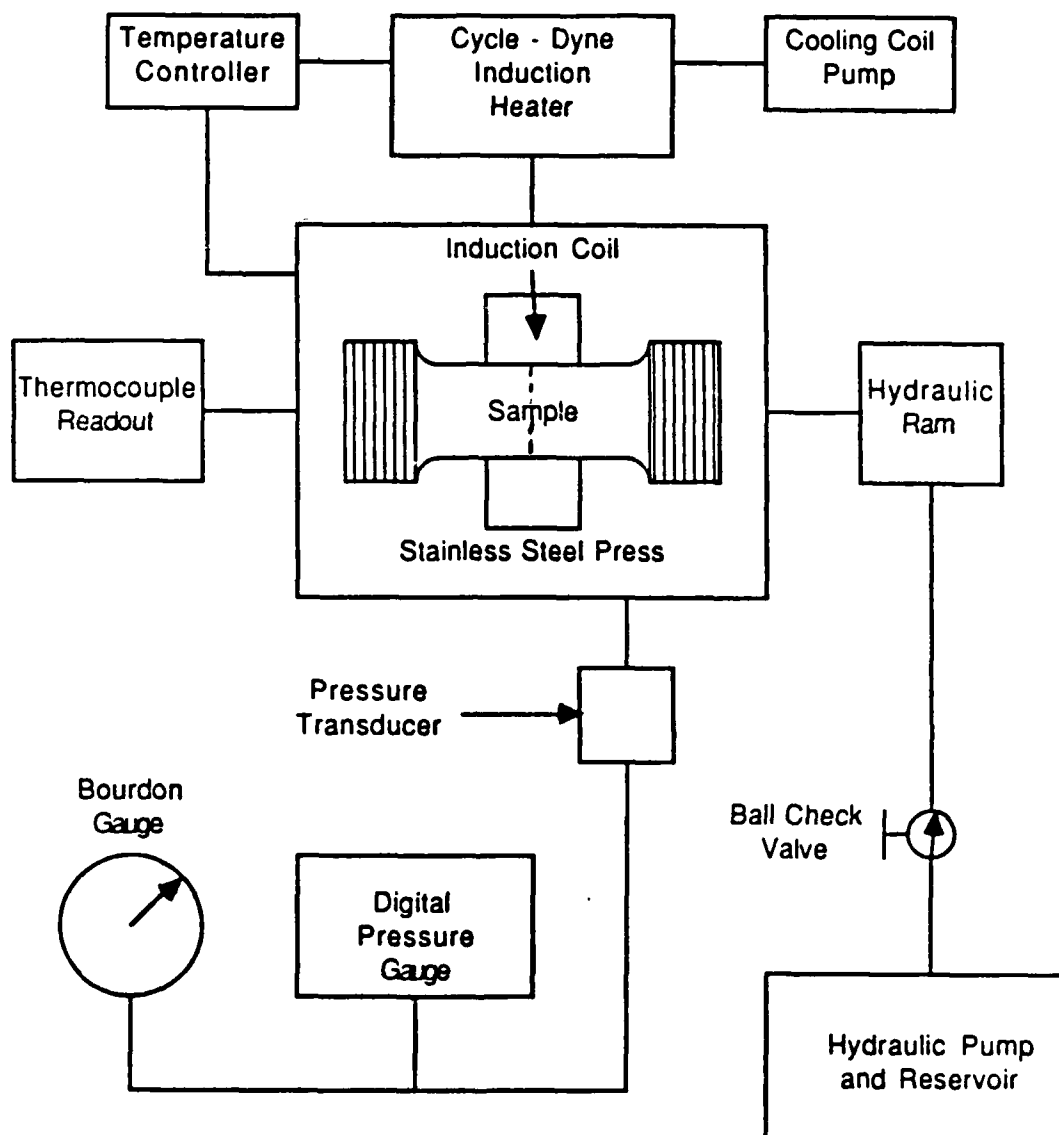
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Figure 3.2 Diffusion Bonding Apparatus Schematic.

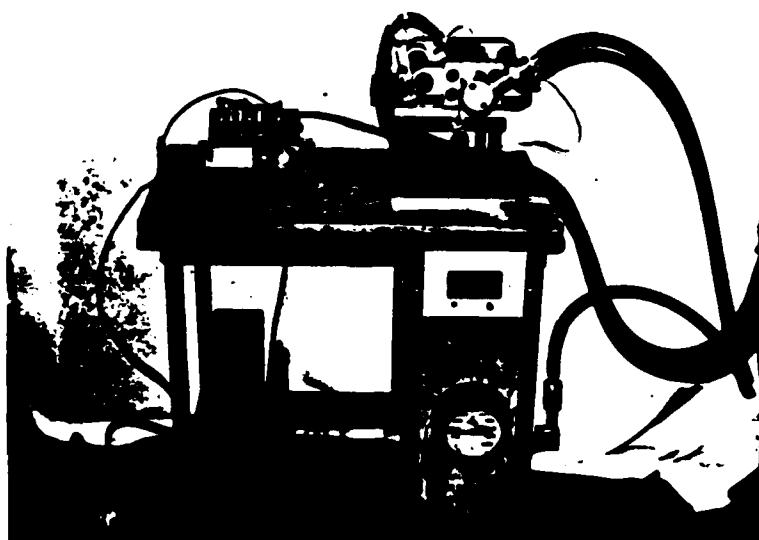Figure 3.3   Diffusion Bonding Apparatus.

Figure 3.4   Stainless Steel Bonding Press.

heating method reduces the chances of an oxide layer forming on the surface. For this research it was desired that only the interface be heated. The thermal gradient will be discussed further in Procedures.

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The Cycle-Dyne provided the power to the induction coil and the coil is kept cool by the cooling water that is pumped through the copper working coil. The spiral pancake coil (Figure 3.7) was used as the working coil. Desired temperatures are achieved by adjusting the set point on the MTS Temperature Control Panel (Figure 3.6) to a pre-calibrated setting. The temperature controller monitors the sample temperature by a thermocouple spot welded to the sample.

Figure 3.5   Cycle-Dyne Model A-30 Induction Heater.
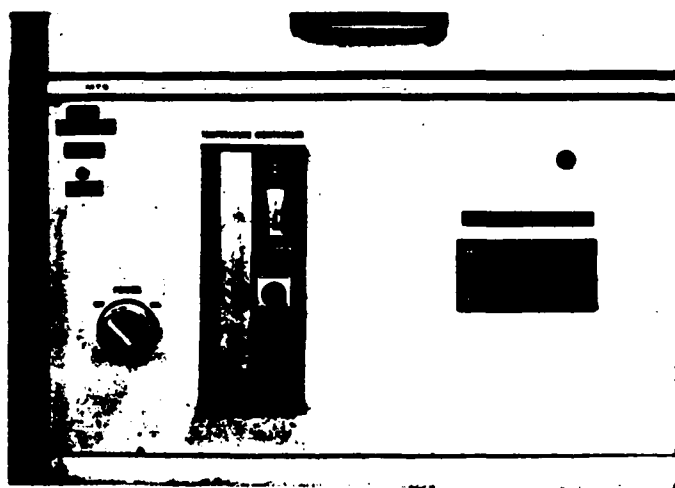

Figure 3.6   MTS Model 924.85 Temperature Control Panel.
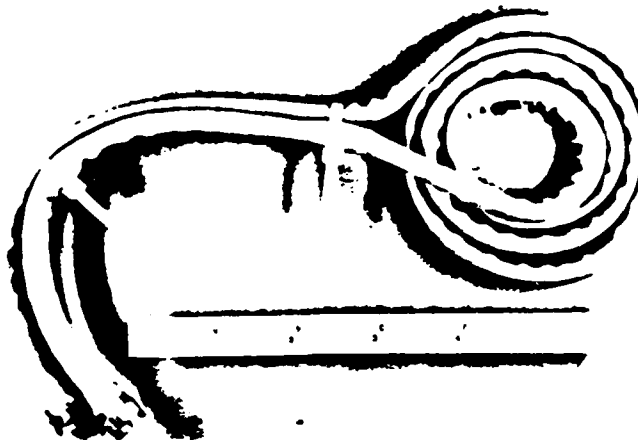
B. EXPERIMENTAL PROCEDURES

Figure 3.7   Spiral Pancake Coil.

1. Preliminary Testing

The checklist of step by step procedures to be followed for performing experiments is listed in Appendix A. Before low temperature bonding experiments were performed, system characteristics were determined. A plain stainless steel sample that had been machined in half was mounted in the press, similar to that shown in Figure 3.8. Eight thermocouples (TC) were attached in the following positions:

1. TC #1 1/2 inch from the left of the interface
2. TC #2 1/4 inch from the left of the interface
3. TC #3 1/4 inch from the left of the interface
4. TC #4 at the interface
5. TC #4a at the interface (MTS Temperature Control)
6. TC #5 1/8 inch from the right of the interface
7. TC #6 1/4 inch from the right of the interface
8. TC #7 1/2 inch from the right of the interface

An induction heating experiment was conducted to determine the temperature gradient present in the sample. The results of the experiment are ploted on the graph

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Figure 3.8 Sample Mounted For Bonding.

in Figure 3.9. The actual interface is marked by zero and 1 unit equals 1/8 inch. The graph shows for each of the digital settings that there was a temperature gradient peak and it was not located at the interface. This was not a major problem. For actual bonding experiments, the temperature of the sample was monitored with only two thermocouples. Each thermocouple was placed 1/8 inch on either side of the interface. The peak temperature was positioned over the interface by adjusting the coil until the two thermocouples displayed the same temperature (or within ± 2°C). This would produce an interface temperatures 12°C over the thermocouple temperatures at 300°C.

Temperature gradient through the thickness of the sample was also measured (Figure 3.10). The data for the figure was produced much in the same way as the previous temperature gradient was determined. Thermocouples were placed radially through the thickness as follows:

1. TC #1 left edge of surface
2. TC #2 1/8 inch left of center
3. TC #3 on center
4. TC #3a MTS Temperature Control
5. TC #4 1/8 inch right of center
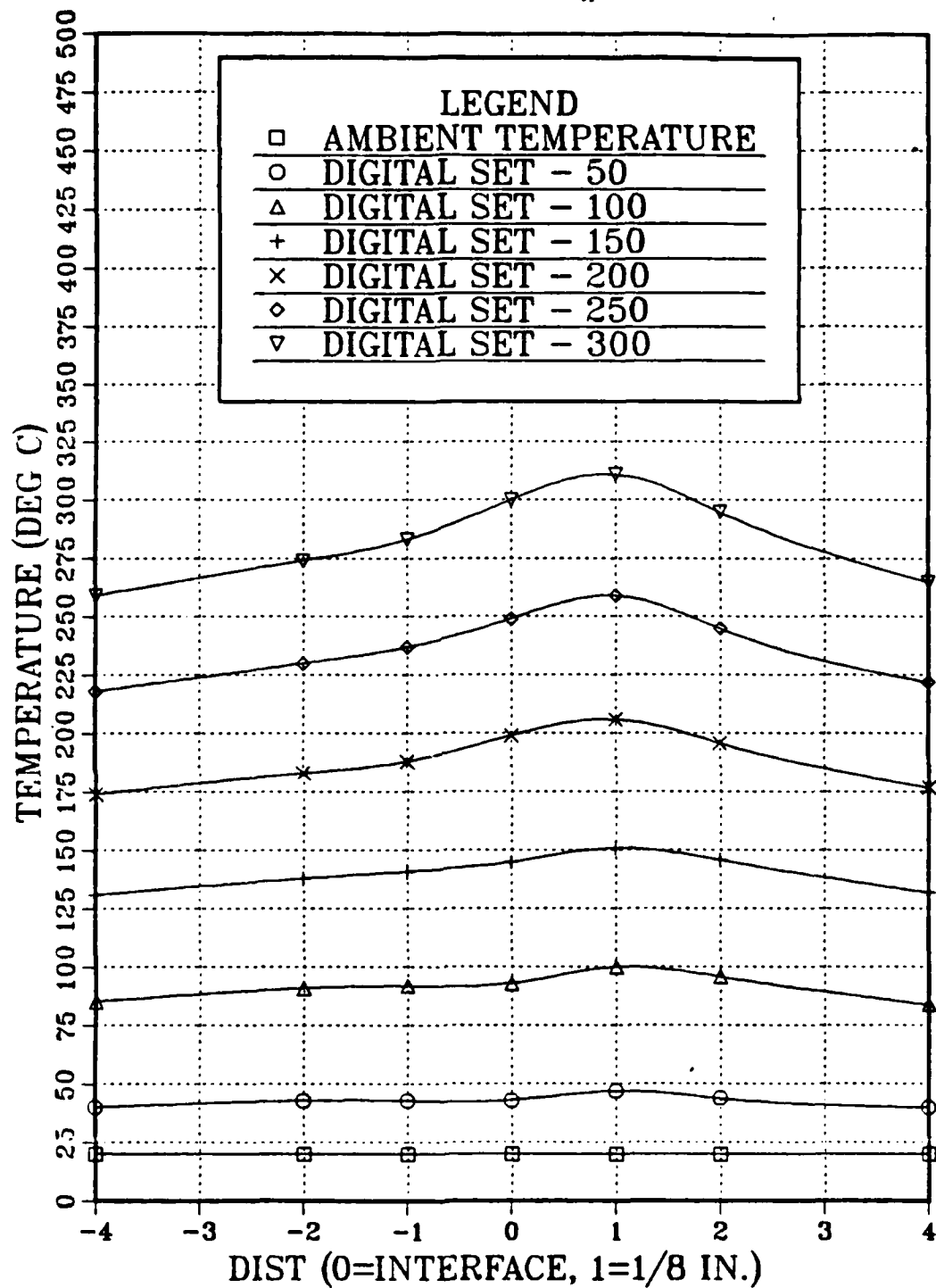6. TC #5 right edge of surface

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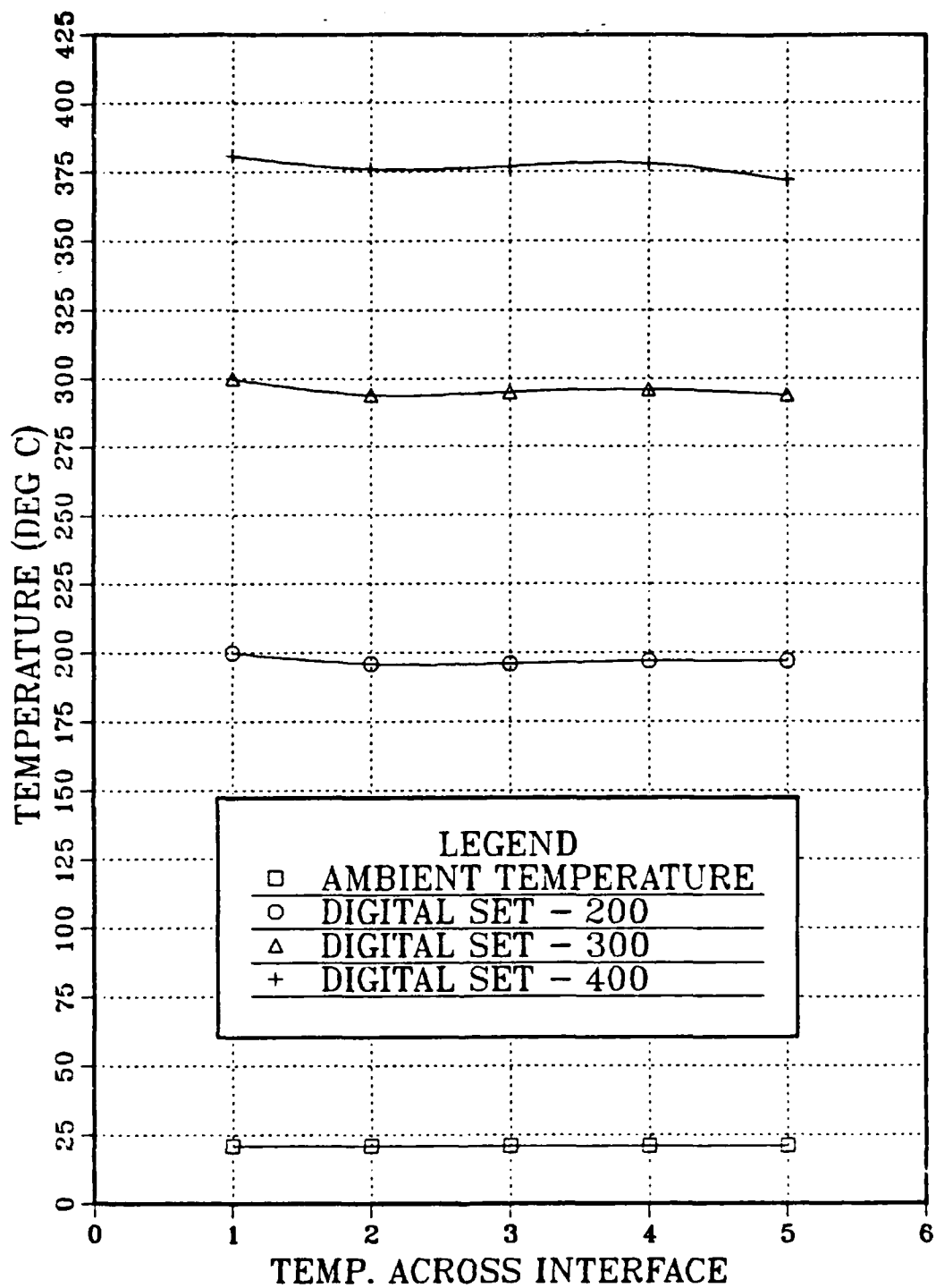Figure 3.9   Spiral Pancake Coil Temperature Gradient.

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Figure 3.10   Temperature Gradient Through the Thickness.

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The data in Figure 3.10 show that practically no temperature gradient exists through the thickness. This result is good and will promote homogenous bonding on the surfaces.

2. Bonding Parameters

The bonding pressure and time were held constant for all bonding experiments. The bonding pressure used was 20 ksi (137.9 MPa) and the bonding time was 30 minutes. These values were chosen to be similar to the experiments performed by: Dini, Kelley, Cowden, and Lopez [Ref. 10].

The temperatures used for the low temperature diffusion bonding were 200°C (473°K), 300°C (573°K), and 400°C (673°K). Table 2 lists the comparison of bonding temperatures to melting temperatures (Tm) [Ref. 14].

TABLE 2

TEMPERATURE COMPARISON

Material	Tm	Bonding Temperature		
		473 K	573 K	673 K
Silver	1233 K	.4Tm	.5Tm	.55Tm
316 Stainless Steel	1648-1673 K	.3Tm	.35Tm	.4Tm

The table shows that the temperatures chosen for these experiments are especially low for the stainless steel.

IV. RESULTS AND DISCUSSION

Throughout the course of this research no diffusion bonds were formed with any significant tensile strength. The lack of diffusion bonding was caused by the fact that the silver coating did not adhere to the stainless steel (SS) substrate. Subsequent analysis by scanning auger analysis indicated that the stainless steel to silver interface was contaminated with hydrocarbons sufficiently to prevent diffusion bonding at this location.

The actual thickness of the silver layer applied (Figure B.1) was 40 microns (1.6 mil). The thickness was four times that required to produce total interlayer thickness of 25 microns (1 mil). Despite the fact that no stainless steel to stainless steel bonds were achieved, there were silver to silver diffusion bonds formed. Figures B.3 through B.8 show the silver interlayer diffusion bonds that were formed at the temperature variations previously mentioned.

Sample B was diffusion bonded at the lowest temperature 200°C. The dark line down the middle of Figure B.3 is the actual interface and the light white lines are the results of interruption sputter coating process. The micrographs in Figures B.3 and B.4 show the very columnar grain structure which is what would be expected from sputter coated silver. Some diffusion bonding did occur across the interface as seen in Figures B.4.

Sample A was diffusion bonded at 300°C. Figure B.2 is a low magnification photograph of an actual bonded region (the lighter colored area around the upper edge) where there was some adherence of the silver to the stainless steel. The two black marks labeled A and B are where the silver-SS interface cracked during the removal of the sample from the press. Micrograph 1 in Figure B.5 shows an area of no bonding, but it is difficult to determine whether or not it was bonded and then separated when pulled apart by the ram during the specimen removal. Micrograph 2 shows the start of recrystallization at the interface and the edges (surface adjacent to the SS). Micrographs 3 and 4 in Figure B.6 show some grain growth across the interface indicative of bonding. Recrystallization and grain growth will be discussed in detail later.

Sample D was diffusion bonded at the highest temperature 400°C. In Figures B.7 and B.8 micrographs 1 and 4 show definite grain growth and recrystallization that has all but obscured the silver to silver interface. Micrographs 2 and 3 show the starting of grain growth at the edges and interfaces. Micrograph 2 shows the possibility of more than one silver to silver interface. The other two interfaces are the result of an interruption in the coating process. When sample D was sputter coated, the process was stopped and there was a 36 hour delay before restart. Vacuum was never broken during this delay, but the cooling down process and restart caused this to be an interface.

The recrystallization temperature for pure is silver around 200°C [Ref. 14]. This temperature is the temperature at which complete recrystallization will occur in a finite period of time usually 1 hour for heavily deformed material [Ref. 15]. The recrystallization temperature is meaningless unless a {inite period of time and the amount of deformation is specified, because recrystallization is affected by time, impurities, and strain or stored energy. The experiments were all conducted at constant time period of 30 minutes and the deformation was minimal, both of which would tend to raise the recrystallization temperature. Since the silver-silver layer is very pure, impurities are not considered a factor in the recrystallization temperature. The driving force for recrystallization comes from the stored energy [Ref. 15]. As an example, two different materials are annealed at the same temperature would result in the material with the larger amount of cold work recrystallizing much faster. This research used constant bonding times for all experiments, therefore the areas with higher stored energy or strain energy will recrystallize first.

In Figure B.5 and B.6 the recrystallization at the silver-silver interface is due to the deformation of the asperities which increases strain energy. Micrograph 2 in Figure B.7 shows three lines of recrystallization. The one in the middle is the silver-silver interface. The other two lines of recrystallization are areas where the sputter coating was interrupted as mentioned earlier. The stopping and restarting of the sputter coating caused smaller grains at the surface. The smaller grains and residual stresses that occur during the initial stages of the coating process when the substrate is cold increased the stored energy and caused this area of recrystallization.

Grain growth follows recrystallization and its driving force lies in the surface energy of the grain boundaries [Ref. 15]. Grain growth can be seen in sample A on the outside edges of micrographs 2, 3, and 4 in Figures B.5 and B.6. At the highest

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temperature, sample D shows major grain growth in micrographs 1 and 4 of Figures B.7 and B.8.

In comparing the results of all the diffusion bonds it is easy to see that the increasing temperature has a major effect on the microstructure. The estimate of the recrystallization temperature is 250 to 300°C. An analysis was attempted to quantify the actual percentage of bonded regions, in lieu of tensile testing, but the analysis was unsuccessful.

V. CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

1. The basic conclusion of this work is that the system and methods developed can perform low temperature diffusion bonding, as noted by the silver to silver diffusion bonds.

2. The failure of the silver layer to adhere to the stainless steel did not effect the results of this research. With properly coated samples, it is assumed a full stainless steel to stainless steel diffusion bond would have been achieved.

3. Results from experimentation show that the temperature variation has a significant effect on the interlayer microstructure. The estimated recrystallization temperature was 250 to 300°C.

4. The diffusion bonding apparatus can be used for further study of the fundamental mechanism of low temperature diffusion bonding.

B. RECOMMENDATIONS

1. Improve the sputter coated silver layers adherence to the samples.

2. Continue research to study the effects of temperature variation on the tensile strengths of the resulting bonds.

3. Conduct experiments bonding dissimilar metals utilizing an deposited interlayer or a thin foil.

APPENDIX A
EXPERIMENTAL SETUP AND PROCEDURES

The purpose of this appendix is to provide a detailed sequence of steps for diffusion bonding samples in the system developed for this research.

1. Check both ground cable leads are securely attached to ground.
2. Check water level in cooling pump reservoir.
3. Check working induction coil for obvious breaks in the insulation and leaks.
4. Start water cooling pump and check for leaks again.
5. Energize Cycle-Dyne by turning on line switch, overload switch, and setting timer switch to off. White light should come on. (Allow for warmup at least 5 minutes).
6. Mount samples in the stainless steel press.
7. Attach thermocouples: a) attach first one 1/8 inch to one side of the interface, b) attach second one 1/8 inch to the other side of the interface.
8. Close ball check valve.
9. Press the samples together using minimum pressure (300-500 psi), (2.1-3.4 MPa).
10. Check sample alignment.
11. Check coil position.
12. Turn on MTS Temperature Control Panel and set digital dial to desired setting.
13. Verify all thermocouples read the same temperature.
14. Start Cycle-Dyne Induction Heating by depressing the blue button. (Red light will come on).
15. NOTE: COIL IS NOW CREATING A RF FIELD TAKE CARE NOT TO SHORT OUT THE COPPER COIL.
16. Record temperature every minute for the first five minutes.
17. Once the temperature has stabilized , adjust coil with nonconductive bakelite to achieve the same temperature readings in thermocouple one and two.
18. After the desired temperature is achieved at the interface, Pressurize the samples together at the desired pressure.
19. Hold samples together for 30 minutes.
20. Record temperatures every 5 minutes until experiment is complete.
21. After the bonding time is up, secure the power to the coil by depressing the red button on the cycle-dyne. (red light should go out).

22. Secure Cycle-Dyne, Water Cooling Pump, and Temperature Control Panel.
23. Allow sample to cool under pressure to approximately room temperature.
24. Slowly release pressure and remove bonded sample.

APPENDIX B
SCANNING ELECTRON MICROSCOPE PHOTOGRAPHS
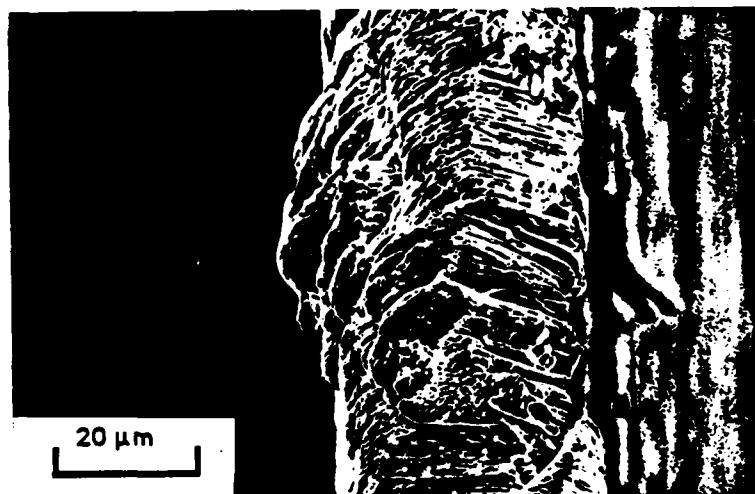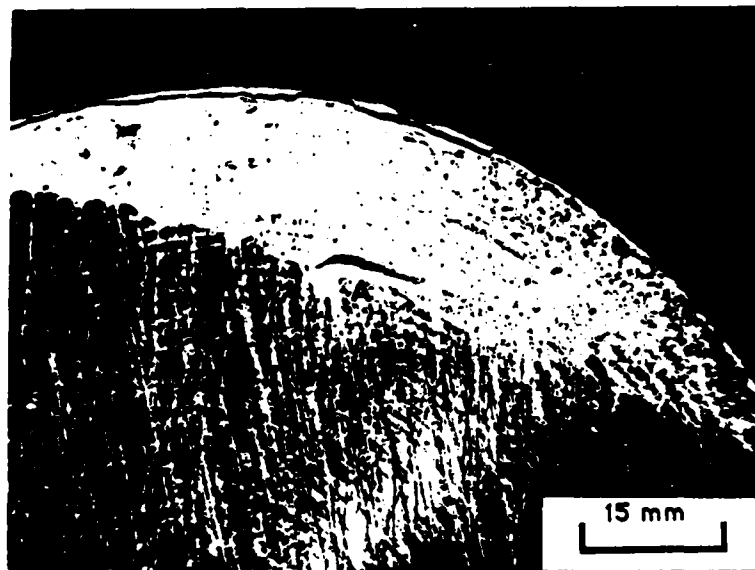

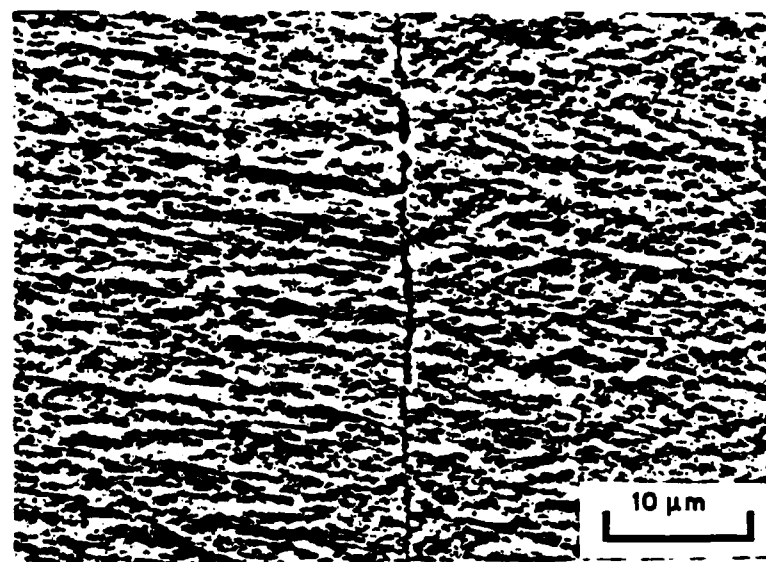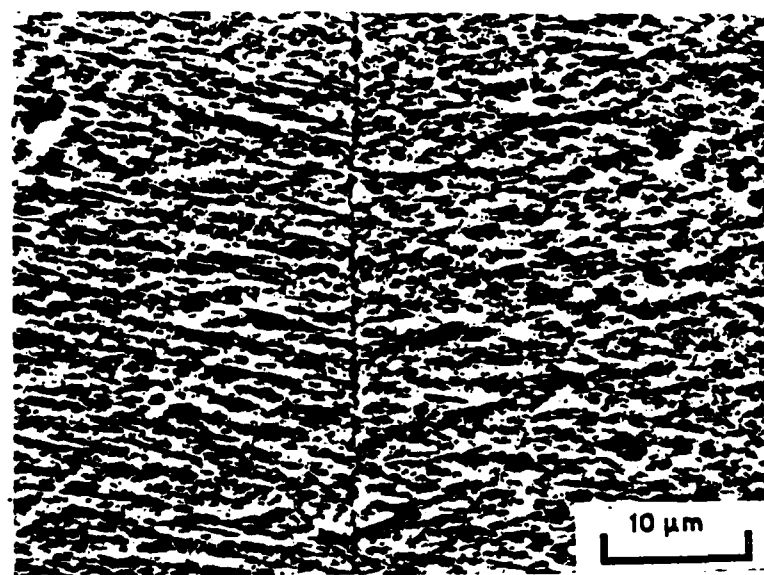Figure B.1   SEM edge view of silver layer.
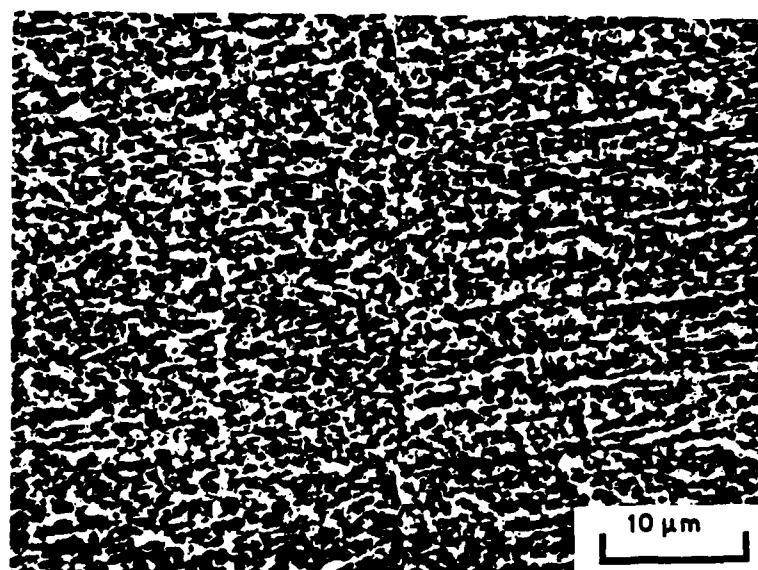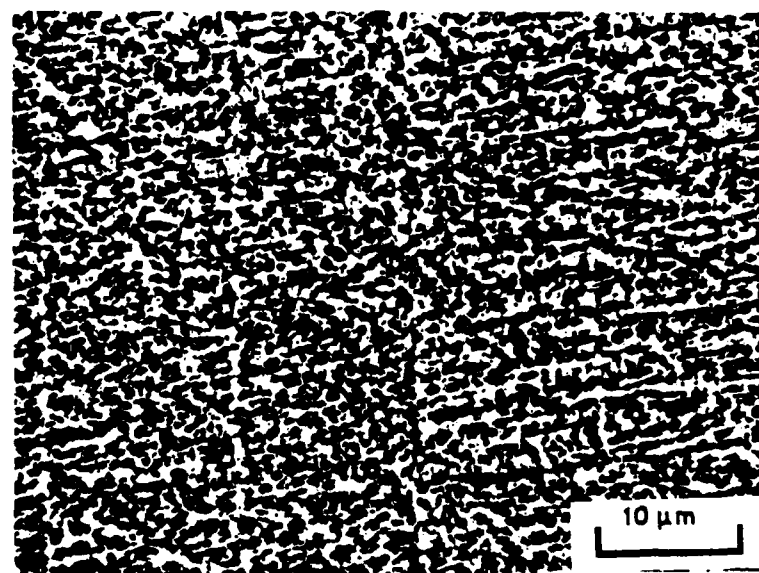

Figure B.2   Bonded surface of sample A.

1

2
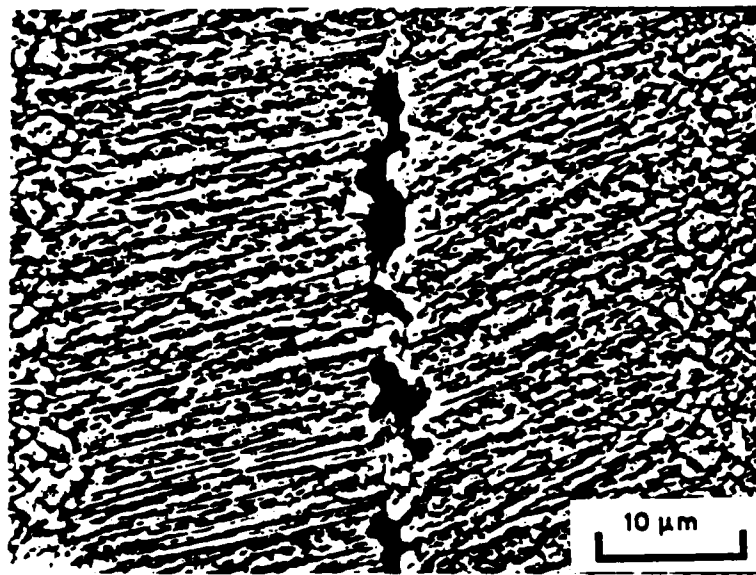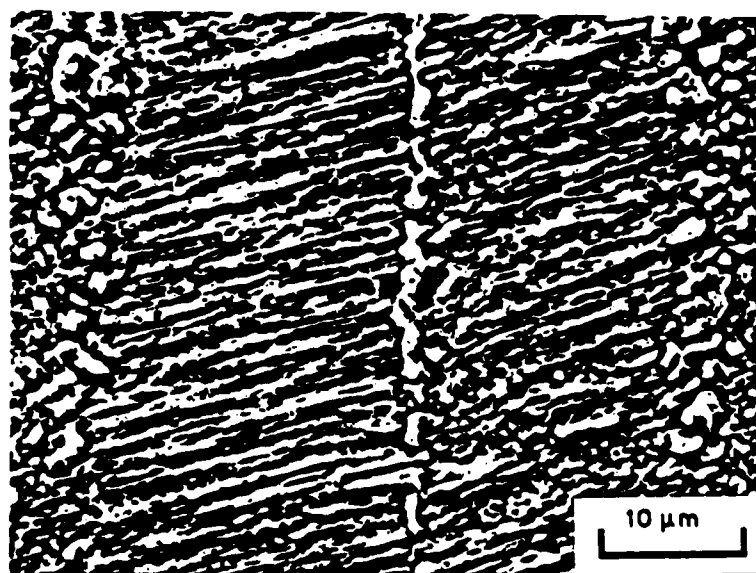
Figure B.3   Sample B silver interface (200° C #1&#2).
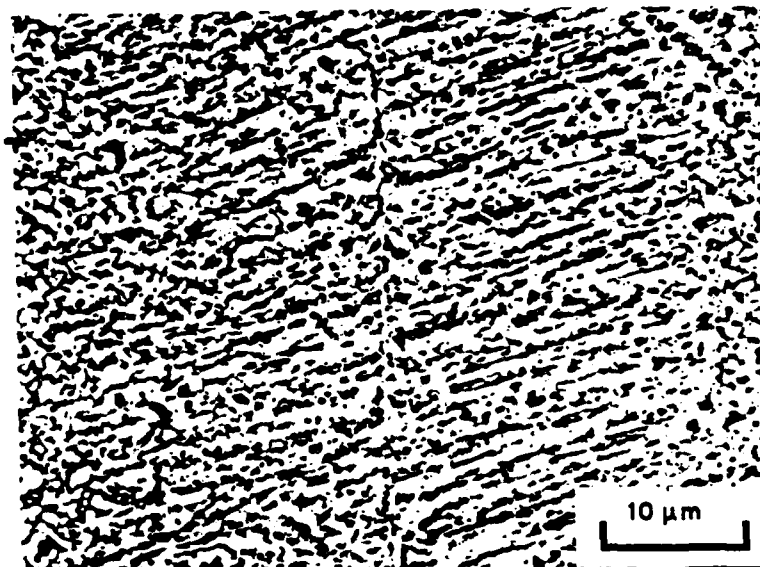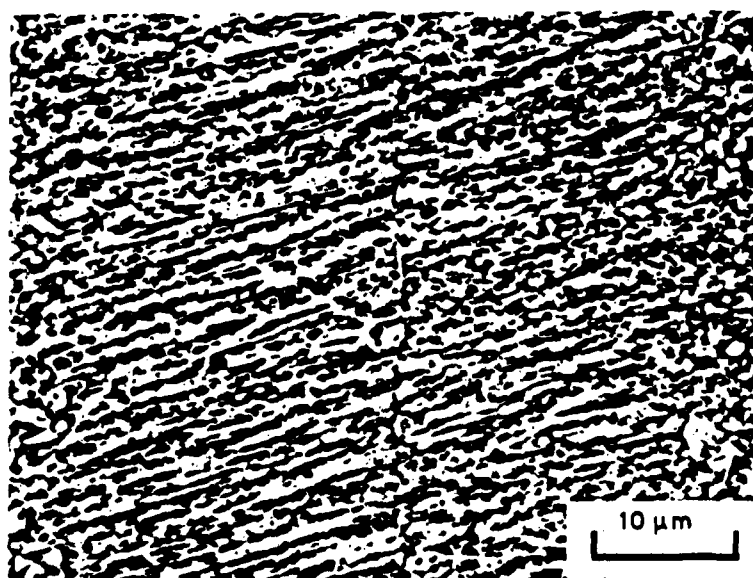
Figure B.4   Sample B silver interface (200° C #3&#4).

Figure B.5   Sample A silver interface (300° C #1&#2).
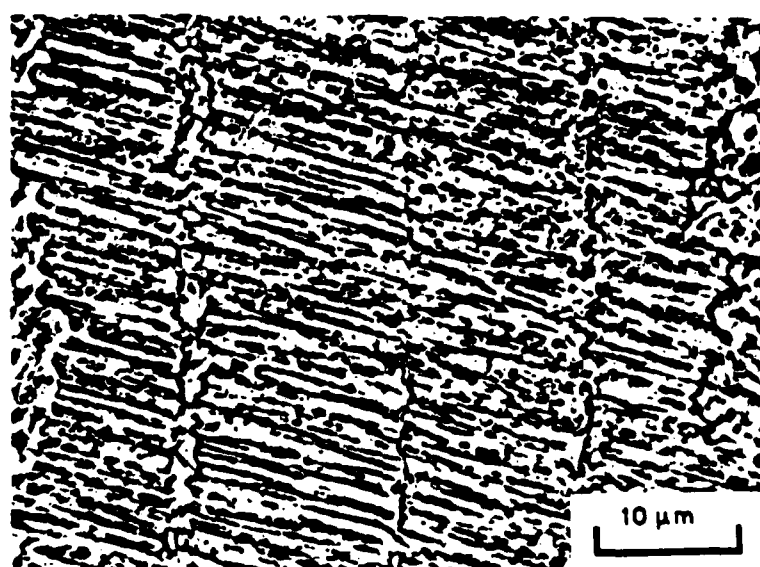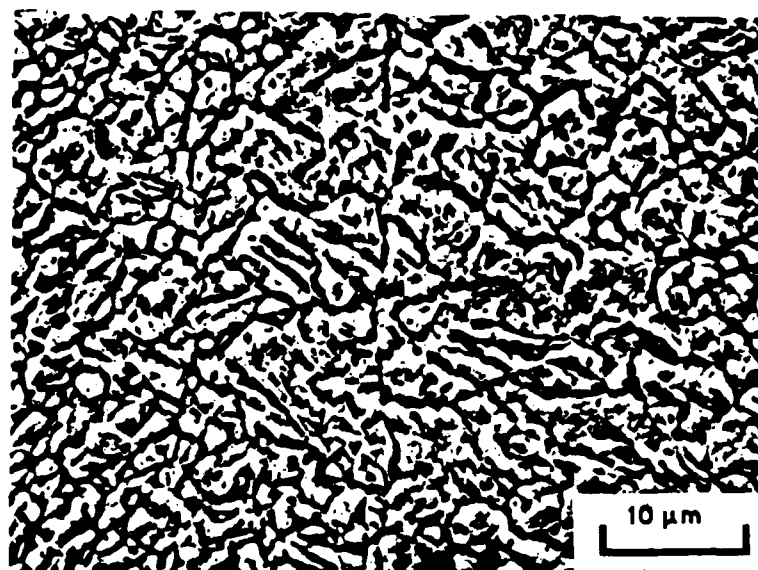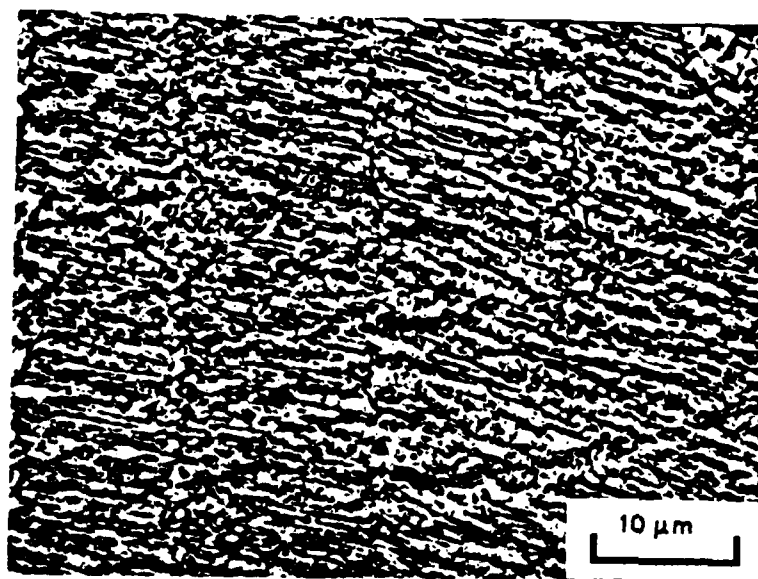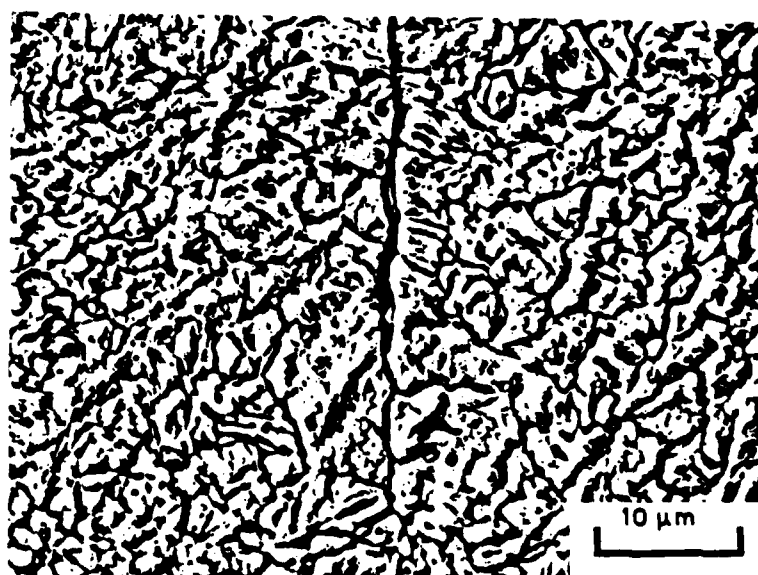
Figure B.6   Sample A silver interface (300° C #3&#4).

Figure B.7   Sample D silver interface (400° C #1&#2).

Figure B.8   Sample D silver interface (400° C #3&#4).

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